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stage is carried out under conditions of backmixing to maintain a level of lower partial fatty acid esters of said polyol is an emulsifying amount; and the final stages are carried out <u>in a continuous manner</u> under plug flow conditions after the degree of esterification is at least about 50%.

In Claim 57, please change "Claim 58" to --Claim 56--.

In Claim 58, line 7, please change "165°C" to --120°C--.

Please cancel Claims 59-61.

REMARKS

Applicants respectfully request reconsideration of the above application, as amended. Claims 1-58 are currently pending.

Enclosed with this amendment is a Supplemental Information Disclosure Statement, together with copies of the documents listed on the attached PTO-1449 form. Applicants respectfully request that the Examiner consider these documents and make them of record in the above application.

The Examiner has rejected Claims 1-61 under 35 USC 112, second paragraph, as being indefinite.

In Claims 1, 22 and 25, the Examiner objects to the term "increasing the mass transfer area of the reaction mixture" as being "functional". This objected-to term also appears in Claims 29 and 35. Applicants submit the Examiner's characterization of this term as "functional" does not render it indefinite when used in the context of the instant Claims. Those skilled in the art, in particular chemical engineers, will understand what is meant by the term "mass transfer area" and in particular what constitutes "increasing the mass transfer area of the reaction mixture". This is particularly shown by the enclosed copies of page 281 from Coulson, <u>Chemical Engineering</u>, (1977), pages 60-63 from Treybal, <u>Mass Transfer Operations</u>, (1980) and pages 360-61 from Westerterp, <u>Chemical Reactor Design and Operation</u>, (1984). Treybal is especially well known among chemical engineers and particularly illustrates what is meant by mass transfer at fluid surface/interfaces like those of the instant Claims, i.e. at the surface/interface between the volatile alcohol and the reaction

mixture.

Moreover, the above application provides a number of representative examples of how the mass transfer area of the reaction mixture can be increased to improve the removal of the volatile alcohol. These representative examples include the use of reactors with larger surface areas, improved agitation of the reaction mixture, sparging of the reaction mixture with an inert gas, or combinations of these methods. See page 4, lines 3-7, and page 18, lines 11-14 and 23-27, of the above applications. Based on these representative examples, one skilled in the art (i.e. a chemical engineer) would understand what is meant by increasing the mass transfer area of the reaction mixture according to the instant Claims, especially in view of Coulson, Treybal and Westerterp. Accordingly, Applicants respectfully request the Examiner to reconsider and withdraw this objection regarding the term "increasing the mass transfer area of the reaction mixture".

In Claim 1, the Examiner has also objected to the term "mixtures thereof" appearing in line 42. Responsive to this objection, line 42 of Claim 1 has been amended to recite <u>combinations of improvements (1) through (10).</u> Support for the underlined language can be found at page 22, line 7, through page 24, line 5, of the above application. This amendment should overcome the Examiner's objection.

The Examiner has also objected to the term "and/or". While not specifying where this term appears in the Claims, Applicants believe the Examiner is referring to Claims 32-33 and 38-39. Responsive to this objection, these Claims have been amended to delete "and/or" and rephrase the claim language. These amended Claims, as rephrased, should overcome the Examiner's objection.

The Examiner has objected to the term "soup" in Claim 53 as lacking antecedent basis. This typographical error has been corrected by changing the word "soup" in Claim 53 to --soap--. Applicants submit there is clear antecedent basis for the term "soap".

In Claim 54, the Examiner has objected to the term "at least" for failing to recite an upper limit. Specifically, Claim 54 recites that Applicants' claimed process is carried out in a series of <u>at least two reaction vessels</u>. Applicants do not understand why the failure to

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recite an upper limit for the number of reaction vessels renders Claim 54 indefinite. The Examiner has given no substantive reason why such a limit is required. As shown at page 21, lines 13-18, of the above application, Applicants clearly contemplate carrying out the reaction in as many reaction vessels as will achieve the objects desired for their claimed process. Accordingly, Applicants respectfully request the Examiner to reconsider and withdraw this objection.

The Examiner has objected to Claims 59-61 on the basis that Claim 60 and 61 are substantial duplicates of Claim 59. Since Claims 59-61 have been cancelled, this objection has been rendered moot.

Beside the amendments responsive to the Examiner's rejection under 35 USC 112, second paragraph, the following additional amendments have been made to the Claims:

a. Claims 1 (see line 13) and 7 have been amended to recite that, when the improvement is a particular level of soap emulsifier, the process is <u>a continuous process</u>. Support for the underlined language can be found at page 2, lines 29-30 and page 22, lines 10-15, of the above application.

b. Claims 1 (see line 25), 12, 19, 42, 52 and 56 have been amended to recite that the molar ratio of total ester reactant to each esterifiable hydroxy group of the polyol is from about 0.9:1 to about <u>1.2:1</u>. Support for the underlined term can be found at page 17, line 11, of the above application.

c. Claims 1 (see line 28), 21, 29, 35, 52, 56 and 58 have been amended to recite that the temperature during the final stages of the reaction is from about 80°C to about <u>120°C</u>. Support for the underlined term can be found at page 18, line 9, of the above application.

d. Claims 1 (see line 30), 22, 25, 29, 35, 52, and 56 have been amended to recite that the pressure <u>in the final stages of the reaction</u> is maintained at from about <u>15</u> to about 300 mm

Hg. Support for the underlined language can be found, respectively, at page 23, lines 7-18 and page 18, line 19, of the above application. The wording in these Claims has also been modified slightly for greater clarity and consistency. The term "interesterification" appearing in Claims 1 (see line 32), 29, 35 and 56 has also been deleted as redundant and potentially confusing, especially in view of the other amendments to these Claims.

e. Claim 1 (see line 36) has been amended to recite that the initial stage of the reaction is carried out under conditions of backmixing. Support for the underlined language can be found at page 19, lines 3-9, of the above application.

f. Claims 1 (see line 38), 16, 48 and 56 (see line 19) have been amended to recite that the final stages of the reaction are carried out in a continuous manner under plug flow conditions. Support for the underlined language can be found at page 23, lines 19-21, of the above application.

g. Typographical errors have been corrected in Claims 1 (see line 11), 43, 53 (see line 4) and 57.

h. The dependency of Claim 54 has been changed from "Claim 1" to "Claim 14".

I. Summary of Applicants' Claimed Improved, Preferably Continuous, Processes for Preparing Highly Esterified Polyol Fatty Acid Polyesters, Particularly Sucrose Polyesters, and the Advantages of these Improved Processes

Applicants' claimed invention relates to improved, preferably continuous, processes for preparing highly esterified polyol fatty acid polyesters, preferably sucrose polyesters, by interesterifying (a) a polyol containing more than 4 esterifiable hydroxy groups and (b) a fatty acid ester of an easily removable alcohol in a heterogeneous reaction mixture, i.e. in the absence of any substantial amount of

unreactive solvent. Applicants' claimed processes involve at least one improvement selected from the following:

(1) The polyol used is a particulate solid, preferably sucrose, that has had its particle size reduced by mechanical size reduction, e.g., grinding, to a particle size of less than about 100 microns, preferably less than about 50 microns, and more preferably less than about 10 microns. See especially Claims 1(1), 2-4, 10-11, and 40-41.

(2) The process is a continuous process in which the initial catalyst level is from about 0.01 to about 0.5 mole, preferably from about 0.01 to about 0.1 mole, most preferably from about 0.02 to about 0.05 mole, of catalyst per mole of polyol. See especially Claims 1(2), 5-6, 10-11, 40-41, 53 and 56.

(3) The process is a continuous process in which the initial level of soap emulsifier in the first stage of the reaction is from about 0.001 to about 0.6 mole, preferably from about 0.2 to about 0.4 mole, per mole of polyol. See especially Claims 1(3), 7-8, 10-11, and 40-41.

(4) After the degree of esterification is greater than about 60% and at least some of any soap emulsifier that is present is insoluble in the reaction mixture, removing the insoluble soap, and any other large particles, by, for example, filtration, in a continuous process. See especially Claims 1(4), 17, 49 and 57.

(5) Any unreacted polyol, e.g., sucrose, and any catalyst having particle sizes above about 1 micron are removed, e.g., by filtration, before the degree of esterification reaches about 75%, and preferably after it has reached 15%, more preferably after 40%, and before any substantial amount of soap emulsifier present has become insoluble in the reaction mixture, preferably in a continuous process. See especially Claims 1(5), 18, 50, 53, 56 and 58.

(6) The molar ratio of total ester reactant to each esterifiable hydroxy group in the polyol is from about 0.9:1 to about 1.2:1, preferably from about 1:1 to about 1.2:1. See especially Claims 1(6), 12-13, 19-20, 42-43, 56 and 58.

(7) The temperature in the initial stage of the reaction is maintained at from about 132°C to about 140°C, preferably from about 132°C to about 135°C, and the temperature in the final stages of the

reaction is from about 80°C to about 120°C, preferably from about 100°C to about 120°C. See especially Claims 1(7), 21, 29-30, 35-36, 52, 56 and 58.

(8) The easily removable alcohol is a volatile alcohol, the reactor pressure in the final stages of the reaction is maintained at from about 15 to about 300 mm Hg, preferably from about 15 to about 100 mm Hg, and the removal of the volatile alcohol produced by the reaction between the ester reactant and the polyol is assisted by increasing the mass transfer area of the reaction mixture, such as by increasing the surface area of the reactor, by sparging with an inert gas such as nitrogen, low molecular weight hydrocarbons, carbon dioxide, etc., to reduce the volatile alcohol's partial pressure, or by combinations of such conditions. See especially Claims 1(8), 22, 25-26, 29, 31-33, 35, 37-39, 52, and 56.

(9) The initial stage of the reaction, and especially in a continuous process that uses multiple reaction vessels, is carried out under conditions of backmixing to maintain a level of lower partial esters of the polyol in an emulsifying amount, typically corresponding to an average degree of esterification of the polyol of from about 10% to about 70%, preferably more than about 20%, more preferably from about 30% to about 60%. See especially Claims 1(9), 14-15, 27-29, 34-35, 46-47, and 54-56.

(10) The final stage, or stages, of the reaction are carried out in a continuous manner under conditions that at least approach plug-flow after the degree of esterification of the polyol has reached at least about 50%, to achieve a final degree of esterification of at least about 70%, preferably at least about 95%. See especially Claims 1(10), 16, 34, 44-45, 48 and 56.

The use of these improvements in an Applicants' claimed processes can lead to a number of significant advantages, including the following;

(a) increased reaction speed and efficiency;

(b) reduced/minimized formation of undesired/unwanted by-products;

(c) reduced/minimized need to remove excess reactants/catalyst;

(d) greater conversion to the desired highly esterified polyol polyester end products;

(e) easier clean up of the desired end products;

(f) reduced/minimized capacity/energy requirements for the equipment used, thus lowering cost and increasing process flexibility.

In achieving these advantages, combinations of these improvements are particularly preferred in Applicants' claimed processes. See page 22, line 6 through page 24, line 5, of the above application.

II. Claims 1-58, as Amended, Which Relate to Improved, Preferably Continuous, Processes for Preparing Highly Esterified Polyol Fatty Acid Polyesters, Particularly Sucrose Polyesters, are Unobvious Under 35 U.S.C. 103 over Volpenhein, even in View of Osipow et al.

The Examiner has rejected Claims 1-58 under 35 U.S.C. 103 as unpatentable over U.S. Patent 4,517,360 (Volpenhein), in view of U.S. Patent 3,644,333 (Osipow et al.). The Examiner says Volpenhein appears to disclose a process closely analogous to that of the instant Claims. The Examiner relies on Osipow et al. to disclose a process for synthesizing sucrose esters, where the reaction occurs between a fatty acid ester and solid sucrose having a very fine particle size (i.e., less than 1 micron in diameter), in the presence of an alkaline catalyst (referring to Column 4, lines 5-9). The Examiner says those Claims related to carrying out the reaction under conditions of backmixing and plug-flow appear to be inherent in the process disclosed by Volpenhein. In conclusion, the Examiner says it would be obvious to modify the process disclosed by Volpenhein to use solid sucrose having the fine particle size taught by Osipow et al. "since the use of sucrose of very fine particle size as shown by Osipow et al. is well known in the art."

For the following reasons, Applicants' respectfully submit the improved processes of the instant Claims, as amended, are unobvious over Volpenhein, even in view of Osipow et al.:

A. Neither Volpenhein, nor Osipow et al., disclose or suggest

using small particle size polyol obtained by mechanical size reduction according to Claims 1 (1), 2-4, 10-11 and 40-41.

One of the improvements used in Applicants' claimed processes is a polyol reactant that has had its particle size reduced by mechanical size reduction to less than about 100 microns. See especially Claims 1 (1), 2-4, 10-11 and 40-41. As taught in the above application, the use of smaller particle size polyol in an esterification reaction to form polyol polyesters is highly desirable to improve the speed of the reaction. This is particularly true in solventless heterogeneous reactions of the type defined in the instant Claims. See page 13, lines 8-14, of the above application.

By contrast, Volpenhein is completely silent as to the particle size of the polyol used in his process. As pointed out by the Examiner, Osipow et al. does teach reacting a fatty acid ester with solid sucrose having a very fine particle size (i.e. less than 1 micron in diameter), in the presence of an alkaline catalyst to form partial sucrose esters, rather than highly esterified polyol polyesters as in the instant Claims. More importantly, Osipow et al. achieves this particle size reduction by dissolving the sucrose in water, rather than by mechanical size reduction as in the instant Claims. Since Osipow et al. needs water to form the microemulsion required by its process, this reference would actually teach away from mechanical size reduction according to the instant Claims.

As pointed out at page 13 of the above application, mechanical size reduction of the polyol according to the instant Claims has a number of significant advantages over particle size reduction achieved by solvents, e.g. by dissolving sucrose in water as in Osipow et al. Although the step of dissolving sucrose in water provides the desired smaller particle size, it also requires the removal of water from the reaction mixture. This usually occurs at a time when other ingredients are present, such that the presence of water can promote the formation of undesirable side products. For example, if water is not completely removed, soap can form during the reaction which reduces the yield of desired highly esterified polyol polyesters and thus increases the cost of the process. Indeed, since catalyst is consumed during this

formation of soap, the reaction may not occur or only at a much slower rate unless more catalyst is added. The presence of a solvent such as water is particularly undesirable in <u>continuous</u> processes.

The solventless mechanical size reduction improvement of the instant Claims has other advantages over particle size reduction achieved by the use of solvents, e.g., water as in Osipow et al. In preparing polyol polyesters, it is highly desirable to avoid exposing the reactants to conditions requiring the removal of solvent such as water, as well as to avoid apparatus/equipment needed for such removal. In addition, to make the process economical, and to avoid excessive losses of the desired polyol polyesters, the solvent may need to be recovered, or if the solvent is water (as in Osipow et al.), the heat used to remove the water (i.e., by evaporation) may also need to be recovered. Additionally, the handling of ingredients while solvent is present undesirably raises the capacity requirements for the process. For example, removal of a solvent such as water can result in a high degree of foaming, particularly if soap is present, unless the solvent is removed carefully and slowly. In summary, the use of smaller particle size polyol obtained by mechanical size reduction according to Claims 1(1), 2-4, 10-11, and 40-41 provides significant advantages in the preparation of polyol polyesters, compared to particle size reduction achieved through the use of solvents, e.g., water as taught by Osipow et al.

Although not relied on by the Examiner in rejecting the instant Claims, Japanese Laid-Open Patent Application 51/14486 cited by Applicants in their Information Disclosure Statement does teach mechanical size reduction of sugar used with fatty acid ester reactants (e.g., methyl esters) to form <u>partial esters</u>. In particular, page 2 of the English translation submitted by Applicants of this Japanese application says the size of the sugar granules should be reduced as much as possible to maximize the surface area and that when the size of the sugar granules is 100 mesh to 150 mesh, this powdered sugar remains suspended in the methyl ester reactant. In addition, page 3 of this English translation says that suitable powdered sugar can be prepared by a conventional grinder, i.e., by mechanical size reduction. However, Applicants believe the sugar particle sizes taught by this

Japanese application would still be larger than the maximum particle size recited in the instant Claims, i.e., less than about 100 microns. This belief is based on the enclosed copies of pages 21-40 and 21-41 from Perry et al., Chemical Engineers Handbook, (5th Edition, 1973) which discloses that 100 mesh and 150 mesh screens provide openings of 149 microns and 105 microns in size, respectively. In other words, the mesh sizes disclosed in this Japanese application suggest this powdered sugar would have all or a substantial percentage of particles greater than about 100 microns in size. (The reference to mesh sizes in this Japanese application typically denotes passage of the particles through the 100 mesh screen and retention on the 150 mesh screen.) Accordingly, Claims 1(1), 2-4, 10-11, and 40-41 are novel and unobvious, even in view of this Japanese Application.

B. Neither Volpenhein, nor Osipow et al., disclose or suggest the use of relatively low catalyst levels in a continuous process according to Claims 1(2), 5-6, 10-11, 40-41, 53 and 56.

Another improvement used in Applicants claimed continuous processes is a relatively low level of catalyst, i.e., below about half a mole, per mole of polyol. See especially Claims 1(2), 5-6, 10-11, 40-41, 53 and 56. It has been surprisingly found that this low level of catalyst still provides fast reactions with little formation of undesired materials. The ability to use relatively low levels of catalyst is particularly important to continuous processes where a long reaction time would be costly in terms of catalyst used. See page 14, lines 11-18, of the above application. Low levels of catalyst are particularly useful in combination with smaller particle size polyol (e.g., sucrose). See Claims 10-11 and 40-41.

Both Volpenhein and Osipow et al. teach using relatively low catalyst to polyol ratios. See Example I of Volpenhein which discloses a potassium carbonate catalyst to sucrose mole ratio of about 0.01. See also Example IV of Osipow et al. which discloses a potassium carbonate to sucrose mole ratio of about 0.02. However, the relatively low catalyst to polyol ratios taught are in the context of a batch reaction, rather than a continuous process. This means neither Volpenhein, nor Osipow et al., recognize the importance of relatively

lower catalyst levels to a continuous process, and accordingly would neither disclose, nor suggest, the processes defined in Claims 1(2), 5-6, 10-11, 40-41, 53 and 56.

At pages 4 to 5 of the Office Action, the Examiner refers in passing to U.S. Patent 4,968,791 (Van der Plank) and U.S. Patent 4,973,682 (Willemse). Applicants would like to point out that each of these references teach the use of relatively low molar ratios of catalyst to polyol, i.e., as low as 0.01:1, preferably from 0.05:1 to 1:1. See column 3, lines 30-32 of Van der Plank and column 4, lines 54-56 of Willemse. However, like Volpenhein and Osipow et al., neither Van der Plank, nor Willemse, teach using low levels of catalyst in a <u>continuous</u> process, as in Claims 1(2), 5-6, 10-11, 40-41, 53 and 56. These references particularly do not suggest the combination of low catalyst levels with smaller particle size polyol according to Claims 10-11 and 40-41. Accordingly, Claims 1(2), 5-6, 10-11, 40-41, 53 and 56 are novel and unobvious, even in view of Van der Plank and Willemse.

C. <u>Neither Volpenhein, Nor Osipow et al., Disclose or Suggest the Use of Relatively Low Soap Emulsifier Levels in a Continuous Process According to Claims 1(3), 7-8, 10-11, and 40-41.</u>

Another improvement used in Applicants' claimed <u>continuous</u> processes is a relatively low initial level of soap emulsifier, i.e. about 0.6 mole or less of soap emulsifier, per mole of polyol. See especially Claims 1(3), 7-8, 10-11, and 40-41, as amended. This relatively low level of soap emulsifier reduces the viscosity of the reaction mixture when the degree of esterification of the polyol has reached at least about 75%. It also reduces undesired losses of the highly esterified polyol polyester end product when the soap emulsifier is removed, as well as reducing excess foaming which can be a particular problem in certain continuous processes when agitation of the reaction mixture is not sufficient, or the pressure under which the reaction is carried out is relatively low. See page 15, line 14, of the above application. Low levels of soap emulsifier are particularly useful in combination with smaller particle size polyol (e.g., sucrose). See Claims 40-41.

Volpenhein and Osipow et al. (see Example IV) both teach using soap emulsifier in their respective processes. However, as pointed out

in part B above, Volpenhein and Osipow et al. are directed at batch reactions, rather than a continuous process. In addition, Volpenhein prefers high soap to polyol mole ratios, i.e. at least about 0.6:1 and preferably from about 0.75:1 to about 1:1 (see col. 3, lines 10-14). Indeed, the disclosures in Volpenhein tend to teach away from the use of relatively low levels of soap emulsifier, and in particular at the preferred mole ratios of the instant Claims, i.e. from about 0.2 to about 0.4. See col. 5, lines 13-18, of Volpenhein where high mole ratios of soap to polyol are characterized as increasing the yield of the highly esterified polyol polyesters, when compared to similar reactions carried out using lower "art-disclosed" soap to polyol mole ratios, e.g., about 0.3-0.4:1. Accordingly, neither Volpenhein, nor Osipow et al., disclose or suggest the use of relatively low soap emulsifier levels in a continuous process according to Claims 1(3), 7-8, 10-11 and 40-41. In particular, these references do not disclose, nor suggest, the combination of low levels of soap emulsifier with smaller particle size polyol according to Claims 40-41.

D. Neither Volpenhein, nor Osipow et al., Disclose or Suggest the Removal of Soap Emulsifier, Especially in a Continuous Process, According to Claims 1(4), 17, 49 and 57

Another improvement used in Applicants' claimed, preferably continuous, processes is the removal of any insoluble soap emulsifier in the reaction mixture (by filtration or centrifugation) after the degree of esterification has reached greater than about 60%. See especially Claims 1(4), 17, 49 and 57. Removal of soap at the early stages of the reaction is not desirable, especially when low levels of soap are used, as is preferred in Applicants' claimed process. However, after the average degree of esterification reaches about 60%, soap is no longer needed to facilitate the reaction and can therefore be removed. Indeed, once the polyol has reacted with the fatty acid esters to provide sufficient lower ester to maintain the homogeneity of the reaction mixture, soap emulsifier is no longer essential. See page 15, lines 29-30, and page 16, lines 5-9, of the above application. Removal of soap can result in reaction mixtures having lower viscosities that provide improved mass transfer of the volatile alcohol

out during the final stages, thus driving the reaction towards completion and leading to shorter reaction times and reduced equipment requirements.

By contrast, Volpenhein and Osipow et al. are completely silent on the removal of soap emulsifier from the reaction mixture. Indeed, neither of these references teach that soap can become insoluble in the reaction mixture, thus making the soap ineffective such that its removal is desirable. Although not relied on by the Examiner in rejecting the instant Claims, European patent application 323,670 cited by Applicants in their Information Disclosure Statement does teach reducing the soap level (such as by filtration or centrifugation) to avoid undesired viscosities in the reaction mixture due to soap separation during the latter stages of the reaction. See page 2, right-hand column, lines 18-23, and 27-43. However, this European application also teaches soap removal at lower degrees of conversion (esterification), i.e., from 15 to 60%, preferably from 20 to 50%, relative to the instant Claims, i.e. greater than about 60%. In addition, this European application does not teach such soap removal in a <u>continuous</u> process. Accordingly, Claims 1(4), 17, 49 and 57 are novel and unobvious, even in view of this European application.

E. <u>Neither Volpenhein, nor Osipow et al., Disclose or Suggest Removal of Unreacted Polyol and/or Large Particle Size Catalyst at an Early Stage of the Reaction in a Continuous Process According to Claims 1(5), 18, 50, 53, 56 and 58.</u>

Another improvement used in Applicants' claimed <u>continuous</u> processes is the removal of unreacted polyol and/or large particle size catalyst at an early stage of the reaction, e.g., before the polyol has been esterified more than about 75%. See especially Claims 1(5), 18, 50, 53, 56 and 58. This removal results in faster reactions kinetics and high conversion to the desired highly esterified polyol polyester end products, with good color and without the need to add additional catalyst thereafter. See page 16, lines 17-20, of the above application.

Removal of unreacted polyol at an early stage of the reaction is more convenient than in the later stages due to the low viscosity of

the reaction mixture and minimizes the formation of unwanted by-products. In particular, unreacted polyol, such as sucrose, can interfere with the orderly progress of the reaction in the later stages by limiting the desired interesterification due to degradation of, and/or preferential reaction with, the active form of the catalyst, by continuing to create undesired by-products such as color bodies, and/or by increasing the time it takes to achieve higher degrees of esterification. See page 16, lines 21-29, of the above application. This is especially true at higher levels of unreacted polyol (e.g., greater than 1% sucrose).

By contrast, Volpenhein and Osipow et al. are completely silent on the removal of unreacted polyol and/or large particle size catalyst, particularly with regard to a continuous process. Accordingly, neither Volpenhein, nor Osipow et al., disclose or suggest this improvement according to Claims 1(5), 18, 50, 53, 56 and 58.

F. <u>Neither Volpenhein, Nor Osipow et al., Disclose or Suggest the Low Ester Reactant to Polyol Molar Ratios According to Claims 1(6), 12-13, 19-20, 42-43, 56 and 58.</u>

Another improvement used in Applicants' claimed processes is a relatively low molar ratio of total ester reactant to each esterifiable hydroxy group in the polyol, i.e. from about 0.9:1 to about 1.2:1. See especially Claims 1(6), 12-13, 19-20, 42-43, 56 and 58, as amended. The advantages of lower ester reactant to polyol molar ratios are that the amount of material in the reaction mixture is less, thus simplifying the processes and permitting the use of equipment with lower capacity for the same output. The risk of undesirable side reactions due to excess fatty acid ester is also minimized. Additionally, control of the degree of esterification is better, and the amount of unreacted fatty acid ester that must be removed is drastically reduced. See page 17, line 11-18, of the above application.

Lower ester reactant to polyol molar ratios also provide considerable improvement in the purification of the end product, without adversely affecting the ease with which the reaction is carried out. Use of these lower ratios of ester reactant to polyol, with the

improvements of lower levels of soap, lower levels of catalyst and/or the removal of unreacted materials before completion of reaction, also leads to end products that can be more readily cleaned up. See page 17, lines 30-37, of the above application. Lower ester reactant to polyol molar ratios are particularly useful in combination with smaller particle size polyol (e.g., sucrose) and/or removal of unreacted polyol. See Claims 12-13, 42-43, 56 and 58).

By contrast, Volpenhein, based on its specific examples, suggests using higher ester reactant to polyol molar ratios in preparing highly esterified polyol polyesters, thus teaching away from the instant Claims. See Example I (two-stage reaction) and Example II (single-stage reaction) where the ester reactant to polyol molar ratio is about 1.6. As pointed out in part A above, Osipow et al. is directed solely at forming partial sucrose esters, rather than highly esterified polyol polyesters as in the instant Claims. Accordingly, whatever teachings there are in Osipow et al. on ester reactant to polyol molar ratios would not suggest the molar ratios of the instant Claims for preparing highly esterified polyol polyesters. In particular, these references do not disclose, nor suggest, the combination of low ester reactant to polyol molar ratios with smaller particle size polyol and/or removal of unreacted polyol according to Claims 12-13, 42-43, 56 and 58.

Applicants would like to point out that Van der Plank and Willemse referred to by the Examiner in the Office Action teach the use of ester reactant to polyol molar ratios as low as 1.25. See col. 3, lines 14-17 of Van der Plank, and col. 4, lines 37-42 of Willemse. However, the lowest molar ratio taught in these references is still higher than that defined in the instant Claims. In addition, Van der Plank and Willemse appear to prefer higher molar ratios based on their specific examples. See Example 1 of Van der Plank where the ester reactant to polyol molar ratio is about 1.8, and Example 2 of Willemse where the ester reactant to polyol molar ratio is about 1.7. Accordingly, Claims 1(6), 12-13, 19-20, 42-43, 56 and 58 are novel and unobvious, even in view of Van der Plank and Willemse.

G. <u>Neither Volpenhein, nor Osipow et al., Disclose or Suggest the Combination of Initial Stage Reaction Temperatures and Lower Final</u>

Stage Reaction Temperatures According to Claim 1(7), 21, 29-30, 35-36, 52, 56 and 58.

Another improvement used in Applicants' claimed processes is an initial stage reaction temperature between about 130°C and 140°C, preferably between about 132°C and about 135°C, and a lower final stage reaction temperature of from 80°C to about 120°C, preferably from about 100°C to about 120°C. See especially Claims 1(7), 21, 29-30, 35-36, 52, 56 and 58, as amended. The reaction temperatures selected in the initial stage achieve rapid initial esterification of the polyol without excessive degradation thereof. However, lower reaction temperatures in the final stages are desirable to minimize side reactions. See page 18, lines 5-10, of the above application. The use of lower final stage reaction temperatures has been surprisingly found to provide a sufficiently fast reaction rate and higher degrees of conversion of the polyol to the desired highly esterified polyol polyester end product, as well as a lighter colored end product.

Volpenhein teaches heating the reaction mixture in the initial stages to temperatures ranging from about 110°C to about 180°C, preferably from about 130°C to about 145°C. Volpenhein also teaches heating this reaction mixture in the final stages (i.e., after excess ester reactant is added) to temperatures of from about 120°C to about 160°C, preferably about 135°C. However, Volpenhein still does not disclose or suggest the combination of initial stage reaction temperatures with lower final stage reaction temperatures according to the instant Claims. This is particularly shown by Example 1 of Volpenhein where the final stage reaction temperature is 135°C, which is much higher than the final stage reaction temperatures now defined in the instant Claims, i.e. maximum of about 120°C.

As pointed out in part A above, Osipow et al. is directed toward forming partial sucrose esters, rather than highly esterified polyol polyesters according to the instant Claims. In other words, Osipow et al. does not have a final reaction stage like that of the instant Claims. Accordingly, Osipow et al. would not disclose or suggest the lower final stage reaction temperatures of the instant Claims.

H. Neither Volpenhein, nor Osipow et al., Disclose or Suggest

<u>the Combination of Higher Pressures During the Final Stages of the Reaction With Increased Mass Transfer Area of the Reaction Mixture According to Claims 1(8), 22, 25-26, 29, 31-33, 35, 37-39, 52 and 56.</u>

Another improvement used in Applicants' claimed processes is the use of higher reaction pressures (from about 15 to about 300 mm Hg, preferably from about 15 to about 100 mm Hg) during the final stages of the reaction, in combination with increasing the mass transfer area of the reaction mixture (e.g., by increasing the surface area of the reactor, by sparging with inert gas, or combinations of such conditions), to improve the removal of volatile alcohol that is produced during the reaction. See especially Claims 1(8), 22, 25-26, 29, 31-33, 35, 37-39, 52 and 56, as amended. Increasing the mass transfer area of the reaction mixture permits the use of these higher reaction pressures while maintaining an acceptable speed of reaction. Use of higher reaction pressures is desirable to avoid having to use sophisticated/costly vacuum equipment, as well as the higher energy requirements required to maintain lower pressures. Higher reaction pressures during the final stages also decrease the formation of undesirable by-products.

Volpenhein does not teach this combination of increasing the mass transfer area of the reaction mixture with higher pressures during the final stages of the reaction. Instead, Volpenhein suggests using lower pressures during the final stages, i.e., from about 0.1 mm to about 10 mm Hg, preferably from about 0.5 mm to about 2 mm Hg. See col. 5, lines 53-60 and step (2) of Example I. Although Volpenhein teaches inert gas sparging to assist in the removal of generated alcohol, it is in the context of using a vacuum, i.e., lower pressures. In short, Volpenhein teaches away from the use of higher pressures during the final stages of the reaction as defined in the instant Claims.

Osipow et al. is completely silent on increasing mass transfer surface area of the reaction mixture. In addition, Osipow et al. teaches the use of <u>lower pressures</u> during their reaction to form partial esters. See Example IV, Col. 7, lines 69-71 where the pressure is 3 mm Hg. Accordingly, Osipow et al. neither discloses, nor suggests, the combination of increased mass transfer area of the

reaction mixture with higher pressures according to Claims 1(7), 21, 29-30, 35-36, 52, 56 and 58.

Although not relied on by the Examiner in rejecting the instant Claims, European patent application 349,059 cited by Applicants in their Information Disclosure Statement teaches using a stripping agent at least during the second stage of the reaction to accelerate the removal of generated alcohol to achieve high degrees (e.g., above 85%) of conversion to sucrose polyesters. (See page 2, lines 46-54.) See also, page 3, lines 2-4, which discloses inert gases, such as nitrogen, as suitable stripping agent. This European application teaches that the pressure during the final stages can be reduced to a level below 50 millibar, preferably to a level below about 25 millibar, such as below 15 millibar, or even below 5 millibar to remove the generated alcohol. See page 3, lines 50-51. In other words, this European application does not teach <u>maintaining</u> higher pressures during the final stages of the reaction, as in the instant Claims. See, in particular, the Example at page 5 of this European application where the pressure is reduced to below 5 millibars (about 4 mm Hg) and then raised to 15 millibars (about 11 mm Hg) when the stripping agent (hexane) is used. Accordingly, Claim 1(8), 22, 25-26, 29, 31-33, 35, 37-39, 52 and 56 are novel and unobvious, even in view of this European application.

I. <u>Neither Volpenhein, nor Osipow et al., Disclose or Suggest Carrying Out the Initial Stage of the Reaction Under Conditions of Backmixing to Maintain an Emulsifying Amount of Lower Partial Esters According to Claims 1(9), 14-15, 27-29, 34-35, 46-47 and 54-56</u>.

Another improvement used in Applicants claimed processes is to carry out the initial stage of the reaction under conditions of backmixing to maintain the degree of esterification between about 10% and about 70%. See especially Claims 1(9), 14-15, 27-29, 34-35, 46-47 and 54-56. This degree of esterification provides sufficient lower partial esters to aid in solubilization of the polyol reactant, as well as a stable heterogeneous reaction mixture that minimizes unreactant polyol and avoids overfoaming of the reaction mixture. See page 19, line 6-12, of the above application. The use of backmixing in the initial stage of the reaction is especially useful in continuous

processes involving multiple reaction vessels. See page 4, lines 8-10, and page 21, lines 13-18, of the above application, as well as Claims 54-55.

At page 4 of the Office Action, the Examiner says backmixing conditions "appear to be inherent in the Volpenhein reference". However, Applicants submit that Volpenhein does not teach backmixing conditions for carrying out his reaction. As taught at the paragraph bridging pages 19 and 20 of the above application, backmixing can be achieved by: (1) continually recycling a portion of the first stage reaction stream; and/or (2) carrying out the reaction in a well agitated vessel (or series of vessels) where the reactants are continually added and the product removed at rates that maintain the desired level of esterification. Volpenhein nowhere discloses either of these methods for carrying out his reaction. That Volpenhein does not disclose backmixing is further shown by the paragraph bridging the enclosed copy of pages 97-98 from Levenspiel, <u>Chemical Reaction Engineering</u> (1972), and especially Figure 1(c) on page 98 referring to a mixed flow (backmix) reactor.

Osipow et al. does teach using a sucrose ester, such as sucrose stearate, as an emulsifier in their partial ester reaction. See col. 4, lines 10-15, 38-42 and 66-72. However, like Volpenhein, Osipow et al. does not teach reaction conditions that inherently involve backmixing according to the instant Claims. Although not relied on by the Examiner in rejecting the instant Claims, Japanese Laid-Open Patent Application 51/14468 cited by Applicants in their Information Disclosure Statement appears to teach the use of sugar esters as an emulsifier medium for its sugar and methyl ester reactants used in preparing partial esters. See page 3 of the English translation of this Japanese application submitted by Applicants. However, like Osipow et al., this Japanese application does not teach reaction conditions that inherently involve backmixing. Accordingly, Claim 1(9), 14-15, 27-29, 34-35, 46-47 and 54-56 are novel and unobvious, even in view of this Japanese application.

J. <u>Neither Volpenhein, Nor Osipow et al., Disclose or Suggest Carrying Out the Final Stage, or Stages, of the Reaction in a</u>

Continuous Manner Under Plug-Flow Conditions According to Claims 1(10), 16, 34, 44-45, 48 and 56.

Another improvement used in Applicants claimed continuous processes is to carry out the final stage, or stages, under plug-flow conditions to achieve high degrees of esterification, e.g., at least about 70%, preferably at least about 95%. See especially Claims 1(10), 16, 34, 44-45, 48 and 56. Plug-flow conditions provide an end product of uniform age and consistency, thus leading to higher degrees of esterification of the polyol. Plug-flow conditions also permit smaller reactor volumes, relative to backmix conditions, for the same degree of esterification.

At page 4 of the Office Action, the Examiner says "plug-flow conditions appear to be inherent in the Volpenhein reference." However, Applicants submit that Volpenhein does not teach plug-flow conditions for carrying out his reaction. As taught at page 21, lines 27-33 of the above application, plug-flow conditions can be approximated by: (1) feeding the output of the initial stage into a series of at least two continuous stirred tank reactors; or (2) preferably and more efficiently, by use of a continuous reactor, for example, a tubular reactor, packed column, tray reactor, or falling/rising film reactor, or combination of such reactors. Volpenhein nowhere discloses these methods, or reactors, for carrying out his reaction. That Volpenhein does not disclose plug-flow conditions is further shown by the enclosed copy of pages 97-98 from Levenspiel, and especially Fig. 1(b) on page 98 referring to a plug-flow reactor.

As pointed out in part A above, Osipow et al is directed toward forming partial sucrose esters, rather than highly esterified polyol polyesters according to the instant Claims. In other words, Osipow et al. does not have a final reaction stage, or stages, like that of the instant Claims. In addition, like Volpenhein, Osipow et al. does not teach reaction conditions that inherently involve plug-flow according to the instant Claims. Accordingly, Osipow et al. would not disclose or suggest the use of plug-flow conditions according to Claims 1(10), 16, 34, 44-45, 48 and 56.

K. <u>Volpenhein and Osipow et al. Especially Do Not Disclose, Nor Suggest, Particular Combinations of Improvements According to Claims 10-18, 21-24, 26-45, 48-53, and 56-58.</u>

As taught at page 22, line 6, through page 24, line 5, of the above application, combinations of improvements are particularly preferred in Applicants' claimed processes. Volpenhein, as well as Osipow et al., especially do not disclose, nor suggest, the following combinations of these improvements;

(a) The combination of (1) smaller particle size polyol obtained by mechanical size reduction, (2) low levels of catalyst, and (3) low levels of soap to provide a fast reaction while minimizing the amount of unwanted materials that are present and that must eventually be removed. See especially Claims 10-11.

(b) The combination of one or more of improvements (a) above with (6) a low ratio of ester reactant to polyol and (9) backmixing to maintain lower partial esters to aid in dissolving the polyol when low levels of soap are used. See especially Claims 12-15, and 27-33.

(c) The combination of improvements (a) and/or (b) above in a continuous process with (4) the removal of soap after the reaction has reached a later, subsequent stage where the soap is no longer needed to dissolve the polyol, is not soluble in the reaction mixture, and can interfere with the reaction rate. See especially Claims 17 and 56.

(d) The combination of (5) polyol removal with improvements (a), (b) and/or (c) above to improve both the speed of esterification and the degree of esterification completeness. See especially Claims 18 and 56.

(e) The combination of (7) lower temperatures and (8) higher pressures in the final stages of the reaction, especially in a continuous process, to allow for fabrication of reaction apparatus without making provision for the maintenance of the more extreme conditions required by prior processes and to allow for savings in energy usage, in addition to the

avoidance of the formation of undesirable and/or unneeded by-products. See especially Claims 22-24, 26, and 29-33.

(f) The combination of (9) backmixing in the initial stage(s) and (10) plug-flow conditions in the final stage(s) is highly preferred to help maintain optimum conditions for initiating the reaction between ingredients that are normally not compatible and then maximizing the final degree of esterification of the polyol. See especially Claims 16, 34, 44-45 and 48.

(g) Combinations of improvement (f) above with improvements (a), (b), (c), (d) and/or (e) above. See especially Claims 35-39, 49-51, and 56-57.

(h) Combinations of (9) backmixing in the first stage(s) and (10) plug-flow conditions in the later stage(s) and (3) low levels of soap emulsifier, and, desirably, (6) low molar ratios of ester reactant to polyol to provide optimum conditions with minimum reactants and to ensure consistent high degrees of esterification in a continuous process. See especially Claims 40-43.

(i) Combinations of (6) low ester/polyol molar ratios, (7) low temperatures; (8) higher pressures; and optionally, but preferably, (5) the removal of unreacted polyol at an early stage of the reaction; and (2) lower catalyst levels to provide low levels of by-products. See especially Claims 52-53 and 56.

III. <u>CONCLUSION</u>

Applicants submit the improved processes of Claims 1-58, as amended, are definite within the meaning of 35 USC 112, 2nd paragraph. Applicants further submit the improved processes of these Claims are unobvious over the references relied on by the Examiner. Neither Volpenhein, nor Osipow et al., disclose or suggest any of the process improvements (1) through (10) defined in these Claims. These references particularly do not disclose, nor suggest, particular <u>combinations of these improvements</u> according to any of Claims 10-18, 21-24, 26-45, 48-53, and 56-58.

Accordingly, Applicants respectfully request that the above application be allowed to issue with Claims 1-58 currently pending.

Respectfully submitted,

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